

PROJECT NUMBER: 2525  
PROJECT TITLE: Tobacco Chemistry  
PROJECT LEADER: S. Tafur  
PERIOD COVERED: July, 1987

#### I. NATURAL PRODUCTS ISOLATION

- A. Objective: To isolate and identify natural products with major emphasis on tobacco and tobacco products.
- B. Results: A Waters photodiode array detector was ordered, received and installed to upgrade our LC capabilities, especially for natural products isolation efforts and analytical LC studies.
- C. Plans: To integrate the new capability into current HPLC work.
- D. References: Core, M. Notebook No. 8417, p.116.

#### II. PROPYL PARABEN

- A. Objective: To find a suitable solvent as a replacement of PG for addition of propyl paraben to the RL process.
- B. Results: An HPLC procedure has been developed for the analysis of paraben methyl or propyl esters and the free acid, p-hydroxybenzoic acid. Using this method a study of the hydrolysis of 10% solutions of propyl paraben in various concentrations (3.4 to 7.4N) of ammonium hydroxide has begun.
- C. Plans: To determine the minimum concentration of ammonium hydroxide necessary to prepare a 10% propyl paraben solution which will last a week without any appreciable hydrolysis.
- D. References: Izac, R. Notebook No. 8379, p.122.

#### III. GREENHOUSE STUDIES

- A. Objective: To maintain the R&D greenhouses, to conduct plant research studies and to provide greenhouse-grown tobacco materials for support of other R&D programs.
- B. Results: A fourth group of Burley 21 plants grown in hydroponic culture has been harvested for Project 1904 in support of the low nicotine program. The 36 plants produced about 12.4 Kg of fresh root tissue (346g/plant). A fifth group of 36 plants is in progress.

The  $^{14}\text{C}$  plant growth chamber has been refurbished with the assistance of A. Burton. A chamber run has begun to produce additional  $^{14}\text{C}$ -labeled bright and burley tobaccos for research purposes.

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The greenhouse phase of the culture x topping study has been completed. All leaves have been oven-cured and will be submitted for chemical analyses.

C. Plans: Continue to provide materials as requested and complete research studies in progress.

D. References: Bass, R. Notebook No. 8495, pp.50-54.

#### IV. SMOKE DISTRIBUTION OF FLAVORS

A. Objective: 1) To demonstrate the differences in smoke distribution for volatile flavors vs. flavor release compounds.  
2) To examine factors which may influence flavor distribution.

B. Results: Comparisons of smokestream distribution of anethole and menthol from menthyl anisyl carbonate (CR-2273) in various cigarette configurations have continued (1). While SS/MS ratios of the flavors from the release compound were similar for standard (KC 137-1) and 12% Mg(OH)<sub>2</sub> papers, the SS/MS ratio decreased in a 40% Mg(OH)<sub>2</sub> model due to both increased MS delivery and lowered SS delivery. The same observation was made for delivery of free anethole in a 40% Mg(OH)<sub>2</sub> model.

As shown previously, paper application of CR2273 along the sideseam of a normal paper leads to very low MS flavor delivery. Since the two sides of Mg(OH)<sub>2</sub> papers are quite different, the effect of applying CR-2273 to the inside vs. the outside of the paper was compared. In both 12% and 40% Mg(OH)<sub>2</sub> models, very low MS flavor deliveries were found by applying CR-2273 to the paper, but with no clear distinction between the sides of the paper. Application of CR-2273 to these papers, however, resulted in a decrease in overall recovery of released flavors. In addition, it was noted that with the Mg(OH)<sub>2</sub> papers the distribution of flavors in SS appeared to shift more to gas phase since a larger amount of flavor collected passed through a Cambridge pad into a back-up cold trap.

An increase in MS delivery of flavors from CR-2273 was shown when used with cigarettes having polypropylene filters. In another attempt to enhance MS flavor delivery from CR-2273, a high loading (20mg/cigt) of the release compound was placed at the front end (1.5cm) of Merit Menthol cigarettes. Neither the first three puffs nor the total MS delivery of anethole or menthol increased (2).

While fairly high levels of CR-2273 have not delivered distinct flavor to MS, a definitive SS anethole odor has been noted as expected.

An attempt to develop an ion chromatographic method for acetic acid in smoke was discontinued due to major interferences in TPM

extracts. Method development has now been focused on a GC method which directly analyzes a TPM extract without acid derivatization. Initial analyses have indicated much lower MS levels than expected while SS levels appear normal (3).

C. Plans: Continue to examine flavor distributions upon smoking.

D. References:

1. Tafur, S. Notebook No. 8490, pp.72-73.
2. Tafur, S.; Grubbs, H.; Levins, R. SS/MS flavor distribution studies: Effect of heavy loading of anisyl menthyl carbonate (CR-2273) on the front end of Merit Menthol. Memo to R. N. Ferguson. 1987 July 20.
3. Keene, C. Notebook No. 8236, p.195.

V. SUPPORT ACTIVITIES

A. Objective: To provide requested assistance for Chemical Research activities.

B. Results: Assistance was provided to Lee Labs in Petersburg on the large scale preparation of poly isopropenyl acetate (1).

Ten handmade cigarettes each in four different cigarette papers were prepared using a blended filler sprayed with  $^{14}\text{C}$ -nicotine (2).

C. References:

1. Izac, R. Notebook No. 8379, p.122.
2. Bass, R. Notebook No. 8495, pp.50-54.

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